(54) **EXHAUST GAS PURIFYING FILTER**

(57) A filter for exhaust gas purification has a honeycomb structure made of a porous ceramic material and having a large number of channels, both given channels at one end of the honeycomb structure and the remaining channels at the other end of the honeycomb structure being plugged so as to be able to use the partition walls of the honeycomb structure surrounding the channels, as a filter layer for exhaust gas, whereas in the thickness of the partition walls is 250 μm or less, the porosity is 40% or more, the average pore diameter is 3 to 7 μm, and the volume of the pores having diameters of 10 μm or more is 20% or less relative to the volume of the total pores. This exhaust gas purification filter has improved trapping efficiency for fine solid particulates of 0.08 μm or less while giving rise to no increase in pressure loss.

FIG.1
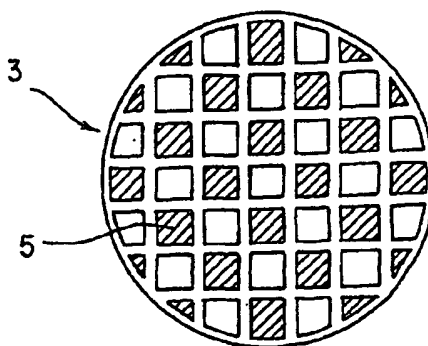
D scription

Technical Field

[0001] The present invention relates to a filter for exhaust gas purification used for removal of solid particulates in exhaust gas, typified by a diesel particulate filter.

Background Art

[0002] In order to remove, from, for example, the combustion gas emitted from a diesel engine, solid particulates composed mainly of carbon, there has been used a filter for exhaust gas purification produced from a honeycomb structure made of a porous ceramic material and having a large number of channels, by plugging given channels at one end of the honeycomb structure and the remaining channels at the other end of the honeycomb structure so as to be able to use the partition walls of the honeycomb structure surrounding the channels, as a filter layer for exhaust gas.

[0003] In such a filter for exhaust gas purification, the relation between the thicknesses of the partition walls of the honeycomb structure (the partition walls function as a filtration layer for exhaust gas layer) and the pore diameters of the partition walls has a large influence on the filter's trapping efficiency for solid particulates. In conventional ordinary filters for exhaust gas filtration, the pore diameters of the partition walls have been about 10 to 20 μm on an average from the property of the solid particulates to be captured, and the partition wall thicknesses have been about 300 to 1,000 μm in view of the pressure loss, strength, etc. of the filter.

[0004] As an example of conventional ceramic honeycomb filters, there is disclosed, in JP-A-56-129020, a ceramic honeycomb filter wherein the given channels of the honeycomb structure are plugged as shown in Fig. 1, at one end of the honeycomb structure and the remaining channels are plugged at the other end and wherein the thicknesses of the partition walls surrounding the channels are 0.1 to 3 mm, the average pore diameter is 10 μm and the porosity is 30 to 60%. In the literature, however, no mention is made on the relation between the partition wall thicknesses and the pore diameters. Also in JP-A-63-185425 is disclosed a ceramic honeycomb filter having partition wall thicknesses of 0.25 to 0.76 mm; however, in this literature, either, no mention is made on the relation between the partition wall thicknesses and the pore diameters.

[0005] Also, in JP-A-5-124021 is disclosed a method for conducting extrusion molding with no deformation or strain by extruding a silicon carbide-based honeycomb into a cooling medium bath. In the literature, the partition wall thickness is set at 0.2 mm; however, no mention is made on the relation between the partition wall thicknesses and the pore diameters. Further, in JP-A-9-202671 is disclosed a method for producing a silicon carbide-based honeycomb filter having partition wall thicknesses of 0.05 to 1.0 mm and an average pore diameter of 1 to 49 μm. In the Examples, there is a description of partition wall thickness = 0.45 mm and average pore diameter = 7 μm; however, in this literature, either, no mention is made on the relation between the partition wall thicknesses and the pore diameters.

[0006] Furthermore, in SAE 950735 is described a cordierite-based honeycomb filter having an average pore diameter of 7 μm; however, the filter has a partition wall thickness of 430 μm and, as a result, give too high a high pressure loss.

[0007] In recent years, there has been a technical progress in diesel engine and, in particular, fuel injection has come to be made at a higher pressure; as a result, the solid particles discharged form diesel engine have become finer and the capturing of such fine solid particles has become a big problem. With conventional filters such as mentioned above, however, there is a fear that, of the solid particles discharged from diesel engine, fine solid particles of 0.08 μm or less blow off the filters.

[0008] In view of the above situation, the present invention aims at providing a filter for exhaust gas purification which is superior in trapping efficiency for fine solid particulates of 0.08 μm or less and which gives rise to no increase in pressure loss.

Disclosure of Invention

[0009] According to the present invention, there is provided a A filter for exhaust gas purification comprising a honeycomb structure made of a porous ceramic material and having a large number of channels, both given channels at one end of the honeycomb structure and the remaining channels at the other end of the honeycomb structure being plugged so as to be able to use the partition walls of the honeycomb structure surrounding the channels, as a filter layer for exhaust gas, wherein the thickness of the partition walls is 250 μm or less, the porosity is 40% or more, the average pore diameter is 3 to 7 μm, and the volume of the pores having diameters of 10 μm or more is 20% or less relative to the volume of the total pores.

Brief Description of the Drawing

[0010] Fig. 1 is a drawing for explaining a state of channel plugging at each end of a honeycomb filter.

5   Best Mode for Carrying Out the Invention

[0011] The filter for exhaust gas purification according to the present invention is produced from a honeycomb structure made of a porous ceramic material and having a large number of channels, by plugging given channels at one end of the honeycomb structure and the remaining channels at the other end of the honeycomb structure so as to be 10 able to use the partition walls of the honeycomb structure surrounding the channel, as a filter layer for exhaust gas. The plugging of channels is preferably conducted by plugging given channels as shown in Fig. 1, at one end of the honeycomb structure and the remaining channels at the other end of the honeycomb structure.

[0012] When an exhaust gas containing solid particulates is passed through one end of such a filter, the exhaust gas flows into the filter through those channels which are not plugged at the one end, passes through the porous 15 partition walls, and enters the channels which are not plugged at the other end. The solid particulates in the exhaust gas are captured by the partition walls when passing through the partition walls, and a solid particulates-removed exhaust gas, i.e. a purified exhaust gas is discharged from the other end of the filter.

[0013] The filter of the present invention is characterized by having a porosity of 40% or more, an average pore diameter of 3 to 7 μm and a volume of pores having diameters of 10 μm or more, of 20% or less relative to the total 20 pore volume. By constituting the present filter as above, the present filter can efficiently capture fine solid particulates of 0.08 μm or less. An average pore diameter of 3 to 6 μm, or a volume of pores having diameters of 10 μm or more, of 10% or less relative to the total pore volume is preferred because it can more efficiently capture solid particles of 0.08 μm or less.

[0014] Further in the present filter, the thicknesses of the partition walls functioning as a filtration layer are set at 250 25 μm or less. Thereby, the present filter can suppress an increase in pressure loss while having excellent trapping efficiency for fine solid particulates of 0.08 μm or less. Wall thicknesses of 150 μm or less are preferred because such thicknesses can show an even lower pressure loss.

[0015] In the filter of the present invention, the honeycomb structure is preferably made of a material selected form the group consisting of cordierite, zirconium phosphate, aluminum titanate, LAS and silicon carbide. Cordierite, zirco- 30 nium phosphate, aluminum titanate and LAS have low thermal expansion coefficients; therefore, use of one material selected from them, as a material for the honeycomb structure can gives a filter superior in thermal shock resistance. When zirconium phosphate, aluminum titanate or silicon carbide is used as a material for the honeycomb structure, a filter superior in heat resistance can be obtained because the material has a high melting point. The plugging agent used for plugging of the channels of the partition walls is preferably made of the same material as for the honeycomb 35 structure because the plugging agent and the material for the honeycomb structure can have the same thermal expansion coefficient.

[0016] The present invention is described in more detail below by way of Examples. However, the present invention is not restricted to these Examples.

40   Examples 1 to 11 and Comparative Examples 1 to 4

[0017] Raw materials for cordierite, i.e. talc, kaolin, alumina, aluminum hydroxide, silica and graphite (their average particle diameters are shown in Table 1) were compounded in proportions shown in Table 1 (the proportion of graphite is relative to the total of the other materials). Thereto were added a binder, a surfactant and water, followed by mixing, 45 to prepare various extrusion-moldable materials. Each material was subjected to extrusion molding to form various honeycomb structures 3 each having a diameter of 144 mm, a length of 152 mm and a partition wall thickness shown in Table 1 and a cell number shown in Table 1. One end of each honeycomb structure was plugged with a plugging material 5 made of the same material as for the honeycomb structure, as shown in Fig. 1 and the other end was plugged so that each channel of the honeycomb structure was plugged at either end. Then, each plugged honeycomb structure 50 was fired at 1,420°C to obtain various filters. Each filter was measured for porosity, average pore diameter, volume of pores having diameters of 10 μm or more relative to total pore volume, initial pressure loss, and trapping efficiency for fine particulates of 0.08 μm or less. The results of the measurements are shown in Table 1.

[0018] Incidentally, porosity, average pore diameter, and volume of pores having diameters of 10 μm or more relative to total pore volume were measured by mercury porosimetry. Initial pressure loss was determined by measuring a 55 difference in pressures before and after filter when the flow amount was 9 m³/min. Trapping efficiency for fine particulates of 0.08 μm or less was determined by measuring, according to a low-pressure impactor method, a difference in particle concentrations before and after filter, for each particle diameter group.

Table 1

| | Raw Materials | | | | | | | | | | | | |
| | Talc | | Kaolin | | Alumina | | Aluminum hydroxide | | Alumina | | Silica | | Graphite (Relative to the total of other components) | |
No.	Average particle diameter (μm)	Batch composition (wt%)	Average particle diameter (μm)	Batch composition (wt%)	Average particle diameter (μm)	Batch composition (wt%)	Average particle diameter (μm)	Batch composition (wt%)	Average particle diameter (μm)	Batch composition (wt%)	Average particle diameter (μm)	Batch composition (wt%)	Average particle diameter (μm)	Batch composition (wt%)
1	5	40	4	10	4	16.5	2	16.5	4	16.5	5	17	40	22
2	7	40	4	10	4	16.5	2	16.5	4	16.5	5	17	40	20
3	5	40	4	10	4	16.5	2	16.5	4	16.5	5	17	40	4
4	5	40	4	10	1.5	16.5	2	16.5	4	16.5	5	17	40	22
5	5	40	4	10	2	16.5	2	16.5	4	16.5	5	17	40	20
6	5	40	4	10	6	16.5	2	16.5	4	16.5	5	17	40	22
7	5	40	4	10	4	16.5	2	16.5	4	16.5	5	17	40	20
8	5	40	4	10	4	16.5	2	16.5	4	16.5	5	17	40	4
9	5	40	4	10	2	16.5	2	16.5	4	16.5	5	17	40	22
10	5	40	4	10	2	16.5	2	16.5	4	16.5	5	17	40	10
11	8	40	4	10	1.5	16.5	2	16.5	4	16.5	5	17	40	10
1	3	40	4	10	4	16.5	2	16.5	4	16.5	5	17	40	20
2	5	40	4	10	1.5	16.5	2	16.5	4	16.5	5	17	40	22
3	5	40	4	10	4	16.5	2	16.5	4	16.5	5	17	-	0
4	5	40	4	10	4	16.5	2	16.5	4	16.5	5	17	40	22

Rows 1–11: Examples. Rows 1–4 (bottom block): Comparative Examples.

Table 1 - Continued

No.	Structure			Properties			
	Wall thickness (μm)	Number of channels (/cm²)	Porosity (%)	Average pore diameter (μm)	Volume of pores having diameter of 10 μm or more (%)	Initial pressure loss (kPa)	Trapping efficiency for fine particles of 0.08 μm or less (%)
1	250	31	52	6	15	1.7	87
2	250	31	51	7	20	1.3	84
3	250	31	41	6	16	5.1	88
4	250	31	50	3	6	9.0	95
5	250	31	49	4	10	5.4	92
6	200	39	53	7	18	0.7	85
7	150	47	50	7	20	0.5	83
8	150	47	40	6	16	2.3	87
9	150	47	50	4	10	1.9	92
10	100	62	45	5	12	1.0	90
11	100	62	45	2	8	2.9	93
1	150	47	51	8	34	0.4	71
2	250	31	48	2	6	14.7	95
3	250	31	36	6	13	9.2	89
4	300	31	52	4	11	10.0	88

Examples: No. 1–11

Comparative Examples: No. 1–4

Examples 12 to 18 and Comparative Examples 5 to 7

[0019]   Two kinds (coarse and fine) of a type SiC materials each having an average particle diameter shown in Table 2 were compounded in proportions shown in Table 2. Thereto were added a binder, a surfactant and water, followed by mixing, to prepare various extrusion-moldable materials. Each material was subjected to extrusion molding to form various honeycomb structures 3 each having a diameter of 144 mm, a length of 152 mm and a partition wall thickness shown in Table 2 and a cell number shown in Table 2. One end of each honeycomb structure was plugged with a plugging material 5 made of the same material as for the honeycomb structure, as shown in Fig. 1 and the other end was plugged so that each channel of the honeycomb structure was plugged at either end. Then, each plugged honeycomb structure was debinded at 400°C in air atmosphere and then fired at a temperature shown in Table 2 in an Argon atmosphere, to obtain various filters. Each filter was measured for porosity, average pore diameter, volume of pores having diameters of 10 μm or more relative to total pore volume, initial pressure loss, and trapping efficiency for fine particulates of 0.08 μm or less, according to the same methods as in Examples 1 to 11 and Comparative Examples 1 to 4. The results of the measurements are shown in Table 2.

Table 2

	No.	Raw Materials				
		SiC coarse particles		SiC fine particles		Firing temperature (℃)
		Average particle diameter (μm)	Batch composition (wt%)	Average particle diameter (μm)	Batch composition (wt%)	
Examples	12	9	80	0.8	20	2200
	13	8	80	0.8	20	2200
	14	8	80	0.8	20	2200
	15	5	80	0.4	20	2150
	16	8	70	0.4	30	2200
	17	5	70	0.4	30	2200
	18	8	70	0.8	30	2200
Comparative Examples	5	11	80	0.8	20	2200
	6	8	80	0.8	20	2100
	7	5	80	0.3	20	2200

Table 2 – Continued

|		Structure		Properties				
	No.	Wall thickness (μm)	Number of channels (/cm²)	Porosity (%)	Average pore diameter (μm)	Volume of pores having diameter of 10 μm or more (%)	Initial pressure loss (kPa)	Trapping efficiency for fine particles of 0.08 μm or less (%)
Examples	12	250	31	48	7	14	2.5	81
	13	250	31	46	6	10	5.3	91
	14	250	31	41	4	7	8.8	93
	15	250	31	43	3	4	9.1	96
	16	150	47	52	7	15	1.9	85
	17	150	47	49	4	5	4.4	94
	18	100	62	50	6	12	0.9	87
Comparative Examples	5	150	47	51	8	24	0.4	74
	6	250	31	38	5	7	9.9	94
	7	300	31	47	4	4	10.8	95

Industrial Applicability

[0020]   As described above, the filter of the present invention has improved trapping efficiency for fine particulates of 0.08 μm or less while suppressing an increase in pressure loss; therefore, can be suitably used as a filter for exhaust gas purification, for example, as a diesel particulate filter.

Claims

1. A filter for exhaust gas purification comprising a honeycomb structure made of a porous ceramic material and having a large number of channels, both given channels at one end of the honeycomb structure and the remaining channels at the other end of the honeycomb structure being plugged so as to be able to use the partition walls of the honeycomb structure surrounding the channels, as a filter layer for exhaust gas,
    wherein the thickness of the partition walls is 250 μm or less, the porosity is 40% or more, the average pore diameter is 3 to 7 μm, and the volume of the pores having diameters of 10 μm or more is 20% or less relative to the volume of the total pores.

2. A filter for exhaust gas purification according to Claim 1, wherein the thickness of the partition walls is 150 μm or less.

3. A filter for exhaust gas purification according to Claim 1 or 2, wherein the average pore diameter is 3 to 6 μm.

4. A filter for exhaust gas purification according to any of Claims 1 to 3, wherein the volume of the pores having diameters of 10 μm or more is 10% or less relative to the volume of the total pores.

5. A filter for exhaust gas purification according to any of Claims 1 to 4, wherein the honeycomb structure is made of a material selected from the group consisting of cordierite, zirconium phosphate, aluminum titanate, LAS and silicon carbide.

FIG.1
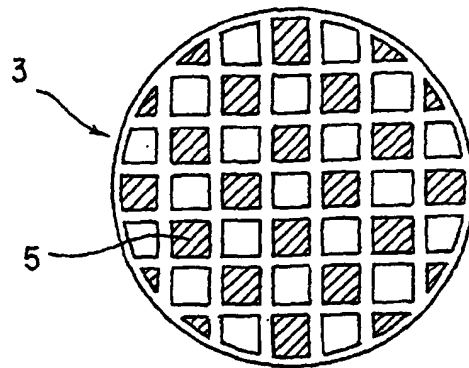
INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl⁷ B01D39/20, F01N3/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
Int.Cl⁷ B01D39/20, F01N3/02

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Jitsuyo Shinan Koho   1926-1996   Toroku Jitsuyo Shinan Koho 1994-2001
Kokai Jitsuyo Shinan Koho 1971-2001   Jitsuyo Shinan Toroku Koho 1996-2001

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
WPI (DIALOG)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP, 9-29024, A (Matsushita Electric Ind. Co., Ltd.), 04 February, 1997 (04.02.97), Claims; Par. Nos. [0011] to [0051] & US, 5846276, A	1~5
Y	US, 5223318, A (Corning Incorporated.), 29 June, 1993 (29.06.93), tables 1,2 & EP, 470340, A1 & JP, 4-231387, A	1~5
Y	JP, 1-224282, A (Inax Corp.), 07 September, 1989 (07.09.89), Claims; page 2, lower left column, line 7 to page 4, upper right column, the last line; Fig. 1 (Family: none)	1~5
A	US, 5549725, A (NGK Insulators, Ltd.), 27 August, 1996 (27.08.96), tables 3,4 & EP, 661088, A1 & JP, 7-163822, A	1~5

☒ Further documents are listed in the continuation of Box C.   ☐ See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed	"&"	document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
06 June, 2001 (06.06.01)	12 June, 2001 (12.06.01)

Name and mailing address of the ISA/	Authorized officer
Japanese Patent Office	
Facsimile No.	Telephone No.

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INTERNATIONAL SEARCH REPORT

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, 5545243, A (NGK Insulators, Ltd.), 13 August, 1996 (13.08.96), Full text & EP, 658363, A1 & JP, 7-163823, A	1-5
A	US, 5069697, A (NGK Insulators, Ltd.), 03 December, 1991 (03.12.91), Full text & EP, 354721, A2 & JP, 2-52015, A	1-5
A	JP, 5-23512, A (IBIDEN CO., LTD.), 02 February, 1993 (02.02.93), Par. Nos. [0005] to [0017] (Family: none)	1-5
A	EP, 834343, A1 (KABUSHIKIKAISHA TOYOTA CHUO KENKYUSHO), 30 September, 1997 (30.09.97), Full text & JP, 10-156118, A	1-5

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